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Effects of deposition temperature and *in-situ* annealing time on structure and magnetic properties of (001) orientation FePt films

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Abstract
FePt films were prepared on (100) oriented single crystal MgO substrates at high temperature ranging from 620 until 800°C and in-situ annealed for different times ranging from 0 to 60 min to obtain ordered FePt films. The structural analysis indicates that FePt films grow epitaxially on MgO (100) substrates. Both increasing deposition temperature and in-situ annealing time enhance the (001) texture and ordering of FePt films. The magnetic analysis shows that these $L1_0$ FePt films have perpendicular anisotropy and the easy magnetization c-axis is perpendicular to the film plane. Magnetization reversal is controlled by a rotational mechanism. The hard magnetic properties of the films are improved with increasing deposition temperature or in-situ annealing time.

Keywords: FePt, texture, hard magnetic, magnetic recording

1. Introduction

The FePt intermetallic alloy with $L1_0$ ordered structure is considered as one of the leading candidate materials for the next generation of ultrahigh-density magnetic recording media because of its large magnetocrystalline anisotropy constant (7×10^7 erg/ cm^3), small grain size (about 3 nm) permitting thermal stability, high coercivity, and excellent corrosion resistance [1]. One of the key challenges of the application of FePt film with $L1_0$ structure for perpendicular magnetic recording media is to control the c-axis texture of FePt films. Usually, FePt films deposited on glass or oxidized Si substrates tend to grow with random orientations or with (111) texture that place the c-axis of grains 37° out of the film plane [2,3]. For perpendicular recording application, the orientation of the $L1_0$ FePt film should have the c-axis perpendicular to the film plane. Many efforts, such as the addition of TiN [4], Ag [5], CrRu [6], CrMo [7], and using RuAl underlayer [8] and nonepitaxial growth [9–13] have been devoted to promoting the $L1_0$ FePt (001) texture as a perpendicular magnetic recording media.

It has also been demonstrated that epitaxial $L1_0$ FePt films with perpendicular orientation of the easy magnetization the c-axis axis have been obtained by growth on single crystal MgO (100) substrates Because the lattice mismatch between $L1_0$ FePt (001) and single crystal MgO (001) is about 8% and the lattice mismatch between Pt(001) and $L1_0$ FePt (001) is 2%, a Pt buffer layer is usually used to reduce the lattice mismatch between $L1_0$ FePt films and single crystal MgO (100) substrates [17,18]. The thermal expansion coefficient of FePt is higher than that of single crystal MgO (100) substrates. The lattice mismatch between L10 FePt (001) and single crystal MgO (001) could be reduced when the films are deposited at high temperature, which means that the deposition temperature can influence the structure and magnetic properties of $L1_0$ FePt films directly deposited on single crystal MgO (100) substrates. In this work, we prepared $L1_0$ FePt films with (001) texture on single crystal MgO (100) substrates and optimized the orientation of the films by controlling the deposition temperature, which could regulate the lattice mismatch between the substrates and the films. The effect of the in-situ annealing time on structure and magnetic properties of (001) orientation FePt films also was studied.

2. Experiments

FePt films with nominal thickness of 5 nm were prepared on (100)-oriented single crystal MgO substrates in an AJA International sputtering system by dc-magnetron and rf-magnetron sputtering at high temperature, ranging from 620 to 800°C. The base pressure of the deposition chamber was about 4.2×10^{-8} Torr, and Ar gas was kept at a pressure of 5 mTorr during sputtering. Before each deposition, the substrate was heated to the deposition temperature for 30 min to prevent moisture and contamination. After each deposition, the sample was also in-situ annealed at deposition temperature for different times ranging from 0 to 60 min to get ordered FePt films. After cooling to room temperature, a 5 nm C layer was deposited on the surface of FePt films to exclude oxidation. The substrates were rotated during film deposition to obtain a uniform film.

The compositions were estimated to be about $Fe_{50}Pt_{50}$ from the sputtering rates of Fe and Pt, which were determined by X-ray reflectivity measurements of the film thickness. The crystal structures of the films were characterized by ϑ–2ϑ X-ray diffraction (XRD) with Cu $K\alpha$ radiation. The magnetic properties were measured with a superconducting quantum interference device (SQUID) up to a maximum applied field of 70 kOe at room temperature.

3. Results and Discussion

3.1. The effect of deposition temperature on structure and magnetic properties

XRD patterns of FePt films deposited at T_d = 620 (a), 700 (b), and 800°C (c) are shown in figure 1. The films were also in-situ annealed at the corresponding deposition temperature for 60 min. The diffraction peaks around at 2ϑ = 23.7° and 48.6° originate from the (001) and (002) reflections of the $L1_0$ crystal structure with (002) a fundamental reflection and (001) a superstructure reflection. Unlabeled peaks in the XRD patterns are from MgO substrates. Only (00n) diffraction peaks are observed in the diffraction patterns, indicating all the FePt films have the (001) orientation and also confirming that the films grow epitaxially on the MgO substrates. The value of the order parameter S of $L1_0$ FePt phase is determined from $0.85 \times [I_{(001)}/I_{(002)}]^{0.5}$, where $I_{(001)}$ and $I_{(002)}$ denote the intensities of (001) and (002) diffraction peaks, respectively [19]. The experimental values for the ratio of the integrated intensities of (001) and (002) diffraction peaks, i.e., $I_{(001)}/I_{(002)}$ have been estimated to be 0.641, and 0.866 for the FePt films deposited at 620 and 800°C, respectively. So the values of the ordering parameter S are calculated to be 0.680 and 0.791, which means that the ordering parameters increase with increasing deposition temperature. A sufficient deposition temperature could promote the Fe and Pt atoms to diffuse during the growth of the film such that they can adopt the correct position in the $L1_0$ phase and obtain higher order parameters. It can also be seen that even though the deposition temperature was increased to 800°C, the films are not still fully ordered. It has been demonstrated that the activation energy of chemical ordering for $L1_0$ phase increases with the decrease of the film thickness, and the ordering temperature rises significantly as the FePt thickness decreases [20]. For our case, the film with nominal thickness of 5 nm is rather thin, so it is hard to get fully ordered $L1_0$ phases, even at 800°C. We also estimated the "perpendicular" grain sizes from the half-peak widths of (001) diffraction peaks, according to the Scherrer formula. The grain sizes along (001) direction are about 7.1, 7.7, and 8.3 nm for FePt films deposited at 620, 700, and 800°C, respectively, which are larger than the nominal thickness of 5 nm. In this work, the volume of magnetic material in the films is estimated from the measured sputtering rates, and the nominal film thickness is 5 nm based on these rates. However, the high deposition temperature causes the Fe-Pt crystals to form islands [21], which causes the packing density to be below 100% and the grain sizes along (001) direction are higher than the nominal thickness of 5 nm.

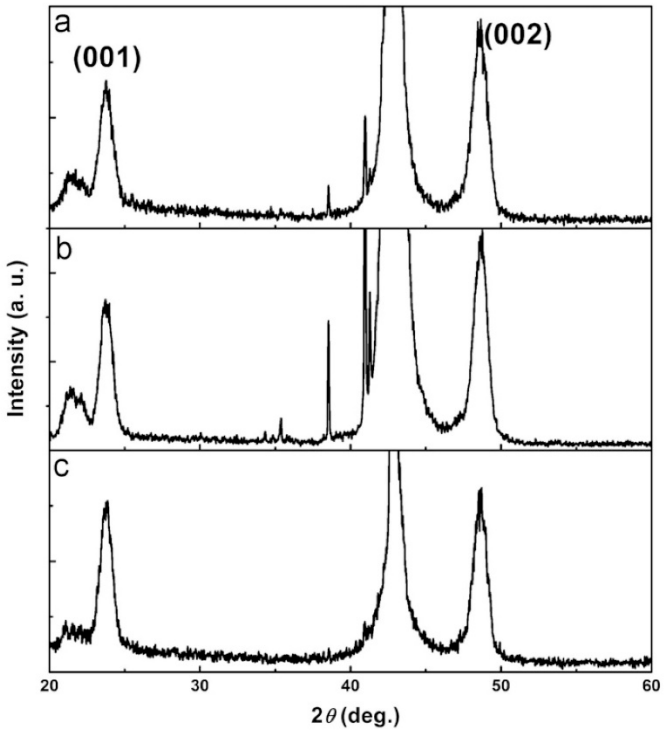

Figure 1. XRD patterns of FePt films deposited at T_d = 620 (a), 700 (b), and 800°C (c).

To further evaluate the degree of misorientation of the films deposited at different temperature, XRD rocking curves for (001) diffraction peaks were measured, and the results are shown in figure 2. The rocking curves can be fitted by a Gaussian function, and the Gaussian half-peak widths ε for the films deposited at different temperature are less than 2.50°, which indicates that the films obtained in the present study are highly (001) oriented. It can be seen that the Gaussian half-peak widths for the films deposited at different temperature decrease with increasing deposition temperature, which means that increasing the deposition temperature could improve the (001) orientation of $L1_0$ phase. Because the lattice mismatch between $L1_0$ FePt (001) and single crystal MgO (100) is about 8% and the thermal expansion coefficient of FePt is higher than that of single crystal MgO (100) substrates, the lattice mismatch between Ll_0 FePt (001) and single crystal MgO (100) could reduce for the films deposited at higher temperature, which could facilitate Ll_0 FePt film growth along the MgO (100) direction and lead to better (001)-oriented FePt films.

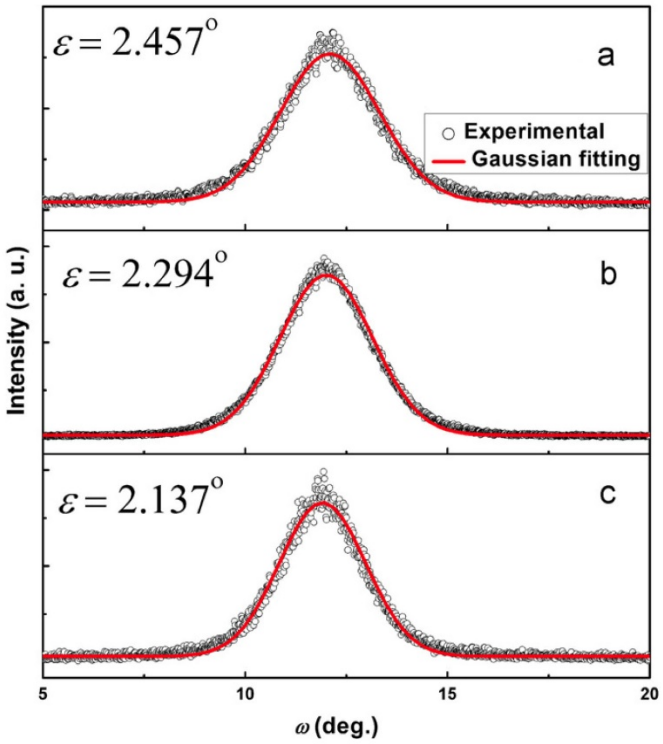

Figure 2. XRD rocking curves for (001) diffraction peaks of FePt films deposited at T_d = 620 (a), 700 (b), and 800°C (c).

Figure 3 shows the in-plane and out-of-plane magnetic hysteresis loops with the initial magnetization curves of FePt films deposited at different temperatures. These show that the easy magnetization c-axis is oriented perpendicular to Ll_0 FePt film plane. It can be seen that the in-plane remanence is also progressively reduced with increasing deposition temperature, suggesting that increasing deposition temperature could enhance the perpendicular anisotropy of the film. The initial magnetization curves of all the films are convex downward and exhibit a critical field value after which the magnetization rapidly increases, which means that the initial magnetization curves have the characteristic of the rotation magnetization because a large magnetic field is required to magnetize the particles [22]. The hysteresis loops were also used to roughly evaluate the effective anisotropy constant expressed as $K_{eff} = H_A M_S/2$, where H_A is the anisotropy field which could be obtained by extrapolating the perpendicular and parallel hysteresis loops, and M_S is the saturation magnetization [23]. The K_{eff} values increase from 3.7×10^7 to 6.9×10^7 erg/cm^3 for the deposition temperature increasing from 620 to 800°C. The out-of-plane coercivities of FePt films also increase from 26.6 to 43.6 kOe when the deposition temperature increases from 620 to 800°C, suggesting that increasing the deposition temperatures could also improve the hard magnetic properties of FePt films.

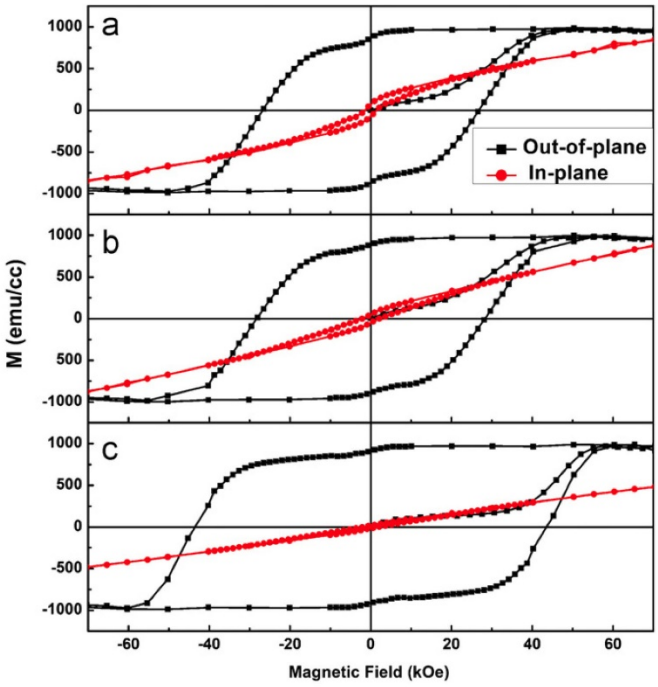

Figure 3. The in-plane and out-of-plane magnetic hysteresis loops with the initial magnetization curves of FePt films deposited at T_d = 620 (a), 700 (b), and 800°C (c).

3.2. The effects of in-situ annealing time on structure and magnetic properties

Figure 4 shows XRD patterns of FePt films deposited at 800°C and in-situ annealed for 0 (a) and 30 min (b). From figure 4, only (001) and (002) diffraction peaks of $L1_0$ FePt phase are seen, which means that FePt film also have (001) texture, even without in-situ annealing. The value of ordering parameters S of $L1_0$ FePt phase in-situ annealed for different time were also calculated. The ordering parameters S are about 0.75 and 0.78 for the films in-situ annealed for 0 and 30 min, respectively. Combined with figure 1(c), it can be seen that increasing in-situ annealing time promotes the ordering of the FePt phase.

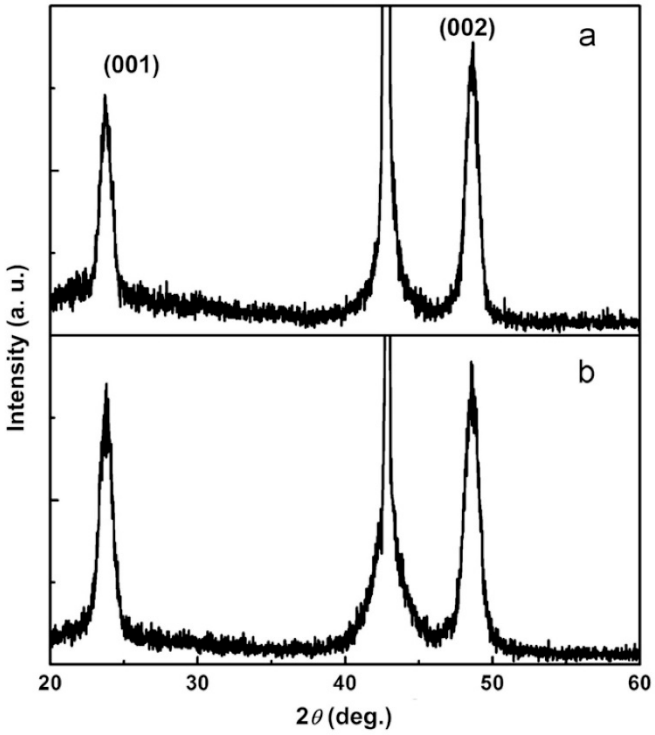

Figure 4. XRD patterns of FePt films deposited at 800°C and in-situ annealed for 0 (a) and 30 min (b).

We also evaluated the misorientation degree of the films deposited at 800°C and in-situ annealed for 0 (a) and 30 min (b) by XRD rocking curves for (001) diffraction peaks, as shown in figure 5. The half-peak widths ε fitted by Gaussian function are less than 2.80°, suggesting that the films in-situ annealed for different time also have high (001) texture. Combined with figure 2(c), we can see that the films have higher (001) texture with increasing in-situ annealing time.

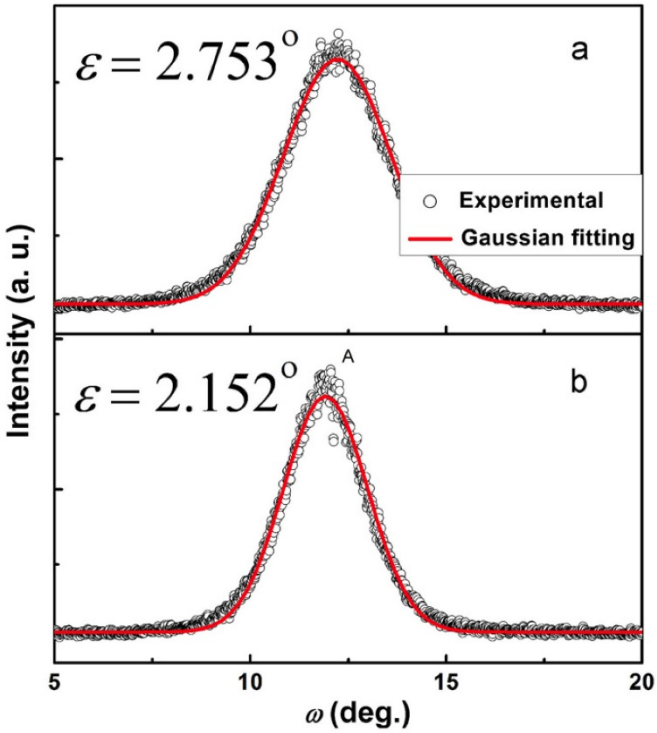

Figure 5. XRD rocking curves for (001) diffraction peaks of FePt films deposited at 800°C and in-situ annealed for 0 (a) and 30 min (b).

The in-plane and out-of-plane magnetic hysteresis loops with the initial magnetization curves of FePt films in-situ annealed for 0 (a) and 30 min (b) are shown in figure 6. From figure 6, we can see that the in-plane magnetic hysteresis loops are not saturated and the out-of-plane hysteresis loops are saturated at a field under 60 kOe, which means that the films have perpendicular anisotropy and the easy magnetization c-axis is perpendicular to $L1_0$ FePt film plane. There is also a critical field around 28.5 kOe in the initial magnetization curves, as shown in figure 6. Below the critical field, the magnetizations of the films increase slowly with increasing the external magnetic field. When the external magnetic field is higher than the critical field, the magnetizations of the films jump up rapidly, which is suggestive of a rotational magnetization mechanism. Apparent shoulders appear in the corresponding out-of-plane hysteresis loops in figure 6, indicating a duplex phase structure due to the coexistence of soft and hard magnetic phases in the films. In our samples, soft magnetic phase should be face-centered cubic FePt phase due to partial ordering. Compared with figure 3(c), we could see that the shoulders almost disappear when the film was in-situ annealed for longer time. This indicates that increasing in-situ annealing time could enhance the hard magnetic properties and ordering of the films.

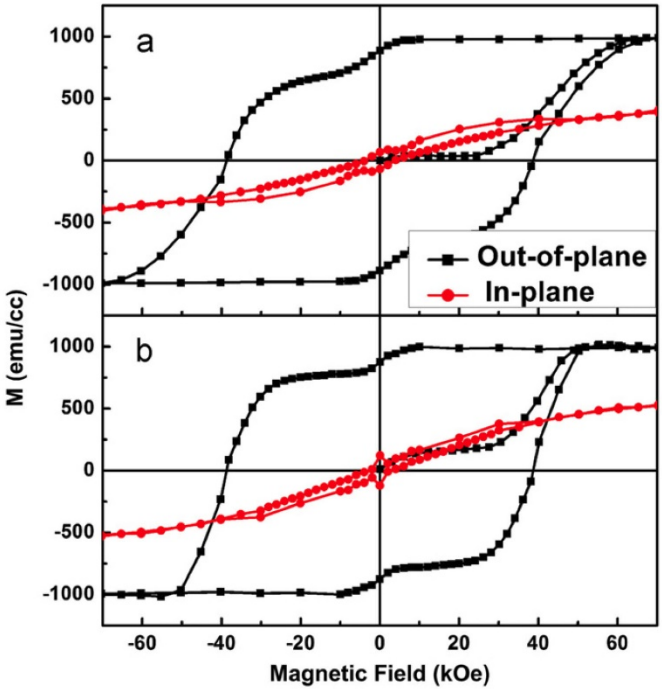

Figure 6. The in-plane and out-of-plane magnetic hysteresis loops with the initial magnetization curves of FePt films deposited at 800°C and in-situ annealed for 0 (a) and 30 min (b).

4. Conclusions

We studied the structure and magnetic properties of FePt films directly deposited on single crystal MgO (100) substrates at high temperature and in-situ annealed for different time. The structural analysis indicates that FePt films grow epitaxially on MgO (100) substrates. (001) texture and order parameters of the films can be enhanced with increasing deposition temperature. The results also show that increasing in-situ annealing time could improve the (001) texture and enhance ordering of the films. The magnetic analysis shows that all of the films in the present research have perpendicular anisotropy and the easy magnetization c-axis is perpendicular to $L1_0$ FePt film plane. Both increasing the deposition temperature and in-situ annealing time enhance the hard magnetic properties of the films.

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